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A COMPOSITION FOR IGNITION MOTORS(U) FOREIGN TECHNOLOGY 1/1  
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FTD-ID(RS)T-1659-82

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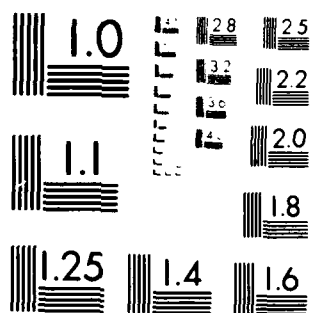


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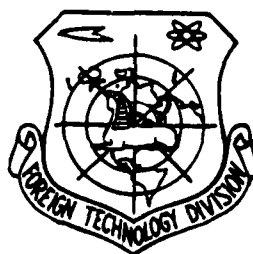
# FOREIGN TECHNOLOGY DIVISION



A COMPOSITION FOR IGNITION MOTORS

by

H. Seimixu, H. Marusen, et al



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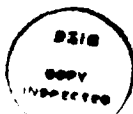
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FTD-ID(RS)T-1659-82

## EDITED TRANSLATION

FTD-ID(RS)T-1659-82

7 January 1983

MICROFICHE NR: FTD-83-0-000017

A COMPOSITION FOR IGNITION MOTORS

By: H. Seimixu, H. Marusen, et al

English pages: 14

Source: Japanese Kokai Patent No. SHO 56(1981) -  
109889, 31 August 1981, pp. 493-498

Country of origin: Japan

Translated by: SCITRAN

F33657-81-D-0263

Requester: FTD/IQTR

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PREPARED BY:

TRANSLATION DIVISION  
FOREIGN TECHNOLOGY DIVISION  
WP. AFB, OHIO.

FTD-ID(RS)T-1659-82

Date 7 Jan 19 83

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19. Japan Patent Office (JP)  
12. Kokai Patent Report (A)  
11. Fokai Patent No. Sho 56(1981)-109889

/493\*

43. Publication Date: August 31, 1981  
21. Application No.: Sho 55(1980)-12376  
22. Application Date: February 6, 1980  
51. Int. Cl.<sup>3</sup>: C 06 B 29/22  
Sequence Nos. for Office Use: 6464-4H..  
number of inventions: 1  
demand for examination: not requested  
(6 pages total)

54. Composition for Ignition Motors

72. Inventors:  
Harusho Seimizu  
8 Taketoyo-cho Nishimon, Chita-gun,  
Aichi-ken  
Harushice Marusen  
4-2-16 Sayama, Sayama-shi  
Shinobu Matsuoka  
7-16 Takiyama, Higashi Kurume-shi  
Daishiro Yamashita  
8 Taketoyo-cho Nishimon, Chita-gun,  
Aichi-ken  
Akira Tobishi Yamaboku  
21-1 Taketoyo-cho, Chita-gun, Aichi-ken  
71. Applicants:  
Nippon Oils and Fats Co., Ltd.  
10-1 Arimoto 1 chome, Chiyoda-ku,  
Tokyo  
Nissan Motor Co., Ltd.  
2 Takashi-cho, Karagawa-ku, Yokohama-shi  
74. Agent:  
Patent Attorney Akedo Sugimura  
1 other person

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\*Translator's note: numbers in margins indicate foreign page numbers.

## Specifications

### 1. Title of the Invention

Composition for Ignition Motors

### 2. Scope of the Patent Claim

1. a composition for an ignition motor which is characterized by the fact that a composition for ignition motors, which is made from a binder, oxidizing agent, combustion agent, and combustion catalyst, contains 58-82 wt% ammonium perchlorate as the oxidizing agent, 7-3 wt% boron as a combustion agent, and 7-3 wt% aluminum as the combustion agent.

### 3. Detailed Explanation of the Invention

This invention pertains to an improved combustion for ignition motors which is used in solid fuel rockets. In further detail, this invention pertains to a composition for ignition motors with a high generation of heat, high combustion rate, and low pressure (illegible) which is characterized by the fact that it contains a mixture of a special oxidizing agent and a special combustion agent.

Heretofore, with regard to spark plugs for solid fuel rockets, priming powder, which has been made into pellets after fuel, such as (illegible), aluminum, etc., and an oxidizing agent, such as (illegible) nitrate are mixed at a ratio of about 3:7 and a small amount of binder, such as nitrocellulose, is added, are placed in an ignition chamber. However, with this type of spark plug, there is a limit to the combustion time range. Moreover, there are problems in that either the ignition delay time during combustion is long or the flammability of the priming powder is insufficient. There are special restrictions in the use of large rockets or land-stage rockets. Thereupon, an ignition motor in which a uniform mixture of a binder, oxidizing agent, and other additives was placed in the ignition chamber was developed in order to solve the aforementioned problems. Although flammability was good, regulation of combustion time was simple, and ignition delay time was short with this type of composition, it cannot be said that this composition was sufficient in the required properties of the amount of generated heat, combustion rate, and pressure index. Therefore, an ignition motor composition displaying high performance in special

applications came into demand. That is, conventional compositions for ignition motors consist of, for instance, a binder, which is made by adding a hardening agent and plasticizer to the main synthetic resin component, such as hydroxylated polybutadiene, carboxylated polybutadiene, etc., an oxidizing agent, such as potassium perchlorate, ammonium perchlorate, etc., a combustion agent, such as magnesium, aluminum, etc., and a combustion catalyst, such as iron oxide, a cupric chromic oxide compound, etc. However, with this type of composition, the amount of heat generated is about 1250 cal/g, the combustion rate is about 20 mm/sec, and the pressure index is about 0.6. In contrast to this, the demands from a high performance composition are about 1400 cal/g of heat generated, a combustion rate of about 22 mm/sec or more, and a pressure index of about 0.5 or less. Nevertheless, when the combustion rate of the aforementioned conventional composition for ignition motors is increased from 20 mm/sec to 22 mm/sec or more by adjusting the mixture ratio, there are problems in that the pressure index becomes higher and therefore the high performance in demand is not obtained.

In general, the pressure index is a value that displays the sensitivity of the rocket propellant and ignition motor composition for the combustion pressure. The correlation between the combustion pressure and pressure index can be expressed with

$$P_c = C K_n \frac{1}{1-n}$$

(In the equation  $P_c$  is the combustion pressure,  $C$  is the constant,  $K_n$  is the ratio between the combustion surface area ( $A_b$ ) and the nozzle throat surface area ( $A_t$ ) ( $A_b/A_t$ ), and  $n$  is the pressure index.) According to this equation, for instance, when the pressure index  $n$  is 0.5, the combustion pressure  $P_c$  is proportional to 2 times  $K_n$  and when the pressure index is 0.75, the combustion pressure  $P_c$  is proportional to 4 times  $K_n$ . Consequently, there is concern over explosion of the ignition motor when combustion pressure is more than the established value, even though there is little change in the nozzle throat surface area and combustion surface area. Therefore, a composition for ignition motors with as low a pressure index as possible is in demand. However, no rules establishing a technique for lowering



the pressure index with a composition for ignition motors has been discovered and therefore, it has been extremely difficult to lower the pressure index to, for example, 0.1.

As a result of carrying out earnest studies on a composition for ignition motors in order to solve the aforementioned problems and to obtain the required properties, the inventors completed this invention upon discovering that when boron is contained as a combustion agent in a composition for ignition motors, which uses ammonium perchlorate as the oxidizing agent and aluminum as a combustion agent, a high amount of heat is generated, the pressure index, is not increased, and a high combustion rate is obtained.

That is, this invention pertains to a composition for an ignition motor which is characterized by the fact that a composition for ignition motors, which is made from a binder, oxidizing agent, combustion agent, and combustion catalyst contains 58-82 wt% ammonium perchlorate as the oxidizing agent, 7-3 wt% boron as a combustion agent, and 7-3 wt% aluminum as a combustion agent.

The reason for using ammonium perchlorate as the aforementioned oxidizing agent is that in contrast to the fact that although a high combustion rate is obtained when boron mixes with potassium perchlorate as the oxidizing agent, for instance, the pressure index becomes 0.5 or more, the combustion rate is then increased and the pressure index is 0.5 or less with a mixture of ammonium perchlorate and boron. Moreover, boron and aluminum are used as the aforementioned combustion agents because although the pressure index does not increase, the combustion rate does not increase with a mixture of ammonium perchlorate and aluminum alone, and on the other hand, a sufficient amount of heat is not generated when ammonium perchlorate is mixed with boron alone.

When the aforementioned ammonium perchlorate content is less than 58 wt%, it is insufficient as an oxidizing agent. Moreover, when the ammonium perchlorate content exceeds 82 wt%, the content of other components is reduced accordingly, which has an effect on the properties of the composition for ignition motors and makes manageability of the composition poor. It is particularly preferred that the ammonium perchlorate content be 60-75 wt%.

Moreover, although an average ammonium perchlorate diameter of 10-200  $\mu\text{m}$  is normally used, it is preferred that the ammonium perchlorate be tiny particles of 1-30  $\mu\text{m}$ .

When each of the boron and aluminum combustion agent contents are less than 3 wt%, the desired combustion properties are not displayed. Moreover, when these contents are greater than 7 wt%, there is a problem from the point of manufacturing in that the crude mixture is not fluid and therefore, it is difficult to obtain a uniform mixture. Moreover, even if it is possible to obtain a uniform mixture, an increase in combustion properties is not obtained. The preferred boron and aluminum contents is 4-6 wt%. Moreover, uniform boron and aluminum particles with a diameter of 0.1-10  $\mu\text{m}$  are usually used. 495

Conventional metallic oxides of iron, copper, chromium, etc. are used as the aforementioned combustion catalyst. For instance, ferric oxide, copper-chromium oxide, etc. may be used. However, ferric oxide is preferred. A small particle diameter is preferred for the combustion catalyst. Normally, a diameter of 0.01-10  $\mu\text{m}$  is used. The combustion catalyst is usually 1-8 wt%. 4-7 wt% is preferred.

The aforementioned binder is made from mainly hard synthetic resins with hardening agents, plasticizers and when necessary, other additives. Normally, about 10-20 wt% of the binder is used in the composition for ignition motors.

Liquid polysulfides, diols, triols, hydroxylated polybutadienes, carboxylated polybutadienes, etc. are examples of the aforementioned hard synthetic resins. With regard to the hardening agents, dioxim compounds are used for the aforementioned polysulfides and di- (or tri-) isocyanate compounds are used for the diols, triols, hydroxypolybutadiene, etc. With regard to the bonding agent, imine compounds, or imine compounds and epoxy compounds for the carboxylated polybutadiene, are used. Moreover, dioctyladipate, dibutylphthalate, dioctylphthalate, etc. are used as the aforementioned plasticizer.

The aforementioned composition for ignition motors from this invention displays high performance with about 1400-1500 cal/g of heat generated, a combustion rate of about 23-27 mm/sec, and a pressure index of 0.40-0.49.

Moreover, the composition for an ignition motor is, for instance, molded into the desired shape after a mixture is obtained by weighing out, mixing and blending the raw materials. That is, a uniform substance is obtained through the molding processes of injection molding (or casting), hardening, and finishing. The risk of fires or explosions during the aforementioned mixing process is as great as the load exerted on the mixer or mixture when the viscosity of the mixture is high. Moreover, there is a strong possibility that bubbles will form inside the mold when the viscosity of the mixture is high during the aforementioned molding process. When bubbles are formed inside the composition, the combustion surface area exceeds the established value and combustion pressure quickly increases in accordance with the aforementioned correlation of  $P_c = CK_n \frac{1}{1-n}$ . Moreover, the combustion rate  $V_b$  increases with an increase in combustion pressure  $P_c$  according to the correlation  $V_b = aP_c^{\frac{1}{n}}$  ( $V_b$  is the combustion rate and  $a$  is the constant). Therefore, there is a chance that the ignition motor will explode with a sudden geometric increase in the combustion pressure. Consequently, in order to manufacture a very reliable composition without any dangers during manufacturing and without any bubbles during molding, the aforementioned mixture must have a suitable viscosity. In general, there is a tendency for the viscosity of conventional compositions for ignition motors, in which a large amount of tiny particles have been mixed, to be high. However, with the composition for ignition motors in this invention, the combustion properties mentioned above can be obtained and a viscosity with which safety, manageability, etc. during the mixing and molding processes can be improved, or that is, a viscosity of about 100 kilopoids (KPS) or less at the conclusion of mixing, can be obtained. Moreover, the composition will have sufficient mechanical strength for practical application.

Furthermore, a composition for ignition motors is used after it is placed in an ignition motor equipped with a primary ignition device and the ignition motor is then placed in the ignition section of a solid fuel rocket motor. However, the composition for ignition motors in this invention is sparked by the primary ignition device and has a short ignition delay time

of about 30 m sec. Consequently, reliability of rocket motor ignition is increased, and at the same time, simultaneous ignition of several rocket motors is improved because the ignition delay time is curtailed. In addition, the weight of the ignition motor composition is decreased and the pressure index is reduced because the ignition delay time is curtailed. Therefore, it is possible to reduce the total weight of the ignition motor and thereby improve the mass fraction (propellant weight rocket motor total weight) because the safety factor with regard to pressure resistance of the ignition motor case can be dispensed. 42r

Next, the composition for ignition motors in this invention will be explained in concrete terms with examples and comparative examples. All numbers and % in each example are based on weight.

#### Example 1

The composition for an ignition motor from this invention was made into a mold using the mixture composition shown in Table 1.

That is, binder (A) was obtained by mixing 1200 parts carboxylated polybutadiene as the main binder solvent, 40 parts tris- 1-(2-methyl)ajirijiniru\* phosphine oxide (abbreviated as MAPO below), 10 parts 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylate (abbreviated as EMEQ below), and 30 parts weight dioctyl adipate as the plasticizer (abbreviated as DOA below). Next, a mixture of 400 parts aluminum (mean diameter of 8  $\mu$ m), 400 parts of boron (mean diameter of 1  $\mu$ m), and 480 parts ferric oxide (mean diameter of 0.1  $\mu$ m) was uniformly dispersed in binder (A). Then 5440 parts ammonium perchlorate (mean diameter of 15  $\mu$ m) was added and the mixture was blended in a vacuum for about 30 minutes at approximately 60° C. After this mixture was placed in a molding device with a diameter of 150 mm and length of 150 mm and bubbles were removed in a vacuum, it was returned to normal temperature. Then the mixture was placed into a drying vat at about 60° C and left to dry for 7 days to obtain the desired composition for ignition motors.

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\*Translator's note: term unknown; transliteration of Japanese phonetic characters.

Furthermore, the viscosity of the composition after the aforementioned vacuum mixing was measured with a B viscometer. These results are shown along with the temperature during the measurements in Table 1.

Next, the combustion properties, consisting of combustion rate, pressure index, amount of heat generated, ignition delay time, and spark delay time, and the mechanical strength of the composition for ignition motors made in this way were measured with the following methods and the results are shown in Table 1.

Table 1

	example no. composition and properties	examples				comparative examples		
		1	2	3	4	1	2	3
mixture composition (%)	binder (A)	16	1	-	16	1	18	1
	binder (B)	-	-	14	-	-	-	-
	ammonium perchlorate	68	82	76	68	78	-	71
	potassium perchlorate	-	-	-	-	-	65	-
	aluminum	5	-	3	-	-	-	-
	boron	5	-	3	3	-	17	5
	ferric oxide	6	-	4	-	3	-	5
	cupric oxide	-	-	-	6	-	-	-
combustion properties	mixture viscosity	60.2 (67°C)	96 (63°C)	80 (64°C)	51.8 (65°C)	22.4 (67°C)	840 (60°C)	80.8 (59°C)
	combustion rate 50 kg cm <sup>-2</sup> , mm/sec	26.4	24.3	26.3	23.4	22.2	20.5	24.3
	pressure index	0.45	0.44	0.48	0.44	0.62	0.57	0.45
	heat generated cal/g	1430	1455	1400	1440	1270	1350	1268
	ignition delay time msec	17	16	22	20	42	91	38
	spark delay time msec	410	400	420	450	500	500	480
	maximum stress kg/cm <sup>2</sup>	20.2	19.2	25.2	16.1	20.0	38.	18.5
	maximum stress deformation %	35	34	31	45	31.5	26	32
mechanical strength (25 °C)	modulus of elasticity kg/cm <sup>2</sup>	75	70	96	80	94	116	73

## a) Combustion rate and pressure index

The combustion rate ( $V_b$ ) under each nitrogen gas pressure of 30 kg/cm<sup>2</sup>, 50 kg/cm<sup>2</sup> and 70 kg/cm<sup>2</sup> was measured using a strand with a length of 5mm, width of 5mm and (illegible) of 100 mm cut from the aforementioned molds. The pressure index ( $n$ ) was then computed with the  $V_b - aP^n$  correlation (provided that  $a$  is a constant and  $P$  is the combustion pressure).

## c) Amount of heat generated

The amount of heat generated from the aforementioned samples was measured under a nitrogen gas pressure of 30 kg/cm<sup>2</sup> using a pump-type calorimeter made by Shimadzu.

## b) Ignition delay time

The ignition delay time is a measure of the ignition capability of an ignition motor for rockers. This time is as good as it is short. It was measured in the following way. That is, the mold of the aforementioned composition for an ignition motor was made into a cylindrical grain with an internal diameter of 34 mm, outer diameter of 54 mm and length of 80 mm. This was then placed in an ignition motor with an electric igniter the aforementioned primary ignition device and pressure pick up attached. This ignition motor was then attached to a rocket motor with an internal combustion polybutadiene composite propellant having an internal diameter of 50 mm, external diameter of 150 mm, and length of 500 mm and a pressure pick up. Combustion tests were then carried out. The pressure-time curve of the ignition motor and rocket motor were simultaneously measured with an oscillograph at this time. The time from the point when the combustion pressure of the ignition motor increased until the point when it reached 75% of the maximum combustion pressure of the rocket motor was measured as the ignition delay time.

## d) Spark delay time

The spark delay time is a measure of the flammability of the composition for an ignition motor. The time required to ignite samples when the aforementioned samples were exposed to 20 cal/cm<sup>2</sup>/sec of laser light was measured as the spark delay time.

## e) Mechanical strength

Tension tests were carried out at 25° C with a stretching rate of 50 mm/minute and a 500 mg tension tester using a

dumbbell-like sample with a total length of 125 mm, a width of 25 mm, a thickness of 10 mm, a 50 mm distance between mark lines, a 10 mm width between mark lines and a 12.5 R curve between the grip and the mark line. The maximum stress, maximum stress deformation and modulus of elasticity at this time were measured.

#### Example 2

A composition for ignition motors with the composition in Table 1 and the same components as in Example 1 was made with the same method as in Example 1. Viscosity, combustion properties, and mechanical strength were measured with the same methods as in Example 1. The results are shown in Table 1.

#### Example 3

Other than the fact that the following binder (B) was used in place of binder (A), a composition for ignition motors with the mixture shown in Table 1 was made as in Example 1. The viscosity, combustion properties and mechanical properties at this time were measured with the same method as in Example 1. The results are shown in Table 1.

Binder (B) was obtained by sufficiently mixing 752.6 parts hydroxylated polybutadiene as the main binder material, 53.6 parts isophorone diisocyanate as the hardening agent, 13.4 parts MAPO as the bonding agent, and 300.2 parts DOA as the plasticizer.

#### Example 4

Other than the fact that cupric chromium oxide (mean particle diameter of 0.14  $\mu$ m) was used in place of ferric oxide as the combustion catalyst, a composition for an ignition motor with the mixture composition shown in Table 1 was made with the same method as in Example 1. The viscosity, combustion properties, and mechanical strength at this time were measured with the same method as in Example 1. These results are shown in Table 1.

#### Comparative Example 1

Other than the fact that boron was used as the combustion agent, a composition for ignition motors with the mixture composition shown in Table 1 was made with the same method as in Example 1. The viscosity, combustion properties and mechanical strength were measured with the same method as in Example 1. These results are shown in Table 1. The combustion properties of this composition are worse than those in Examples 1-4.



#### Comparative Example 2

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A composition for ignition motors with the composition shown in Table 1, where binder (A) is used as the binder, potassium perchlorate is used in place of ammonium perchlorate (mean diameter of 15  $\mu$ ), and boron is used as the only other component, was made with the same method as in Example 1. The viscosity, combustion properties, and mechanical strength of the composition at this time were measured with the same method as in Example 1. The results are shown in Table 1. Although combustion rate is sufficient with this composition, the other combustion properties are worse than those in Examples 1-4.

#### Comparative Example 3

Other than the fact that aluminum was used as the combustion agent, a composition for ignition motors with the mixture composition shown in Table 1 was made with the same method as in Example 1. Viscosity, combustion properties, and mechanical strength at this time were measured with the same method as in Example 1. The results are shown in Table 1. This composition displayed inferior heat generation and ignition delay time in comparison to Examples 1-4.

#### Comparative Examples 4-6

Compositions for ignition motors with the mixture compositions in Table 2 were made as in Example 1 with the amount of aluminum, and boron being outside the range given in this invention. Because the amount of aluminum and boron was large in Comparative Example 4, a solid mixture with poor fluidity was obtained when these components were evenly dispersed in binder (A) and the ammonium perchlorate was added. Consequently, it was difficult to mix the composition with a mixer and a uniform mixture could not be obtained. Therefore, the molding process was interrupted. It was possible to obtain the desired compositions with Comparative Examples 5 and 6 without the aforementioned mixing problems. Next, the viscosity, combustion properties, and mechanical strength of comparative Examples 5 and 6 were measured with the same method as in Example 1. These results are shown in Table 2.

Although the desired combustion rate was obtained, index pressure and heat generation were insufficient with Comparative Example 5. Moreover, in the case of Comparative Example 6, the desired pressure index was not obtained as it was a high value of 0.58, even though combustion rate and heat generation were sufficient. In addition, ignition delay time of Comparative Examples 5 and 6 was about 2 times longer than that of Examples 1-4. As can be seen from the aforementioned, when the amount of aluminum and boron mixed is beyond the range of this invention it is difficult to mix the composition and when the amount is less than the range in this invention combustion properties cannot be satisfied.

Table 2

comparative example no.		4	5	6
mixture composition	composition and properties			
	binder (A)	17	17	17
	ammonium perchlorate	63	63	63
	aluminum	8	2	8
	boron	3	2	2
combustion properties	ferric oxide	4	6	5
	mixture viscosity (KPS)	-	(illegible, (56°C)	58 (54°C)
	combustion rate 50 kg cm <sup>2</sup> , mm/sec	-	(illegible)	23.6
	pressure index	-	0.53	0.58
	heat generation cal/g	-	1280	1420
mechanical strength	ignition delay time msec	-	43	36
	spark delay time msec	-	530	470
	maximum stress kg/cm <sup>2</sup>	-	17.5	19.0
	maximum stress deformation %	-	40	38
	modulus of elasticity kg/cm <sup>2</sup>	-	82	97

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